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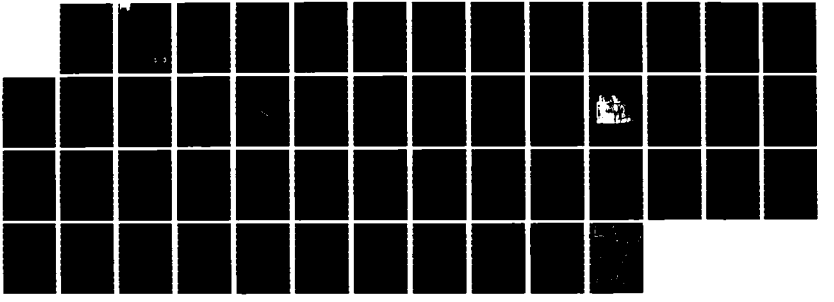

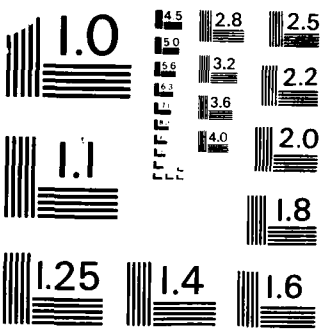

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TECHNICAL REPORT SL-86-36

DEVELOPMENT AND FIELD PLACEMENT OF AN EXPANSIVE SALT-SATURATED CONCRETE (ESC) FOR THE WASTE ISOLATION PILOT PLANT (WIPP)

by

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An expansive salt-saturated concrete (ESC) was proportioned for placement underground in halite rock at the Waste Isolation Pilot Plant (WIPP) site, near Carlsbad, New Mexico. Requirements for this concrete were: (1) to be chemically compatible with the host rock; (2) to remain pumpable for four hours; (3) to give net volume increase beginning at an early age, and continuing until creep closure of the salt assures sealing at the rock interface; and (4) to cure to a solid with extremely low permeability and fairly high strength.

ESC was proportioned and placed underground at the WIPP in two successful field tests during FY 85 and FY 86. This report is the first of three reports about this concrete. It describes (1) the development of ESC in the laboratory, and (2) the mixture properties prior to final set. It summarizes field placement activities in July 1985 and February 1986, when ESC was placed in test holes underground at the WIPP for two series of Small-Scale Seal
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Performance Tests (SSSPT). It also gives data from tests of expansive behavior of the concrete at early ages and under simulated repository conditions.

The second report will describe expansive behavior of ESC relative to several variables that could have an impact on its field performance and long-term stability, as determined during laboratory testing. It also will discuss possible explanations of the rather extraordinary suite of properties exhibited by ESC, as controlled by its chemical composition. The third report will describe laboratory studies of the mechanism of set retardation in a grout derived from this concrete.

PREFACE

The work described in this report is part of an ongoing research effort accomplished in the Concrete Technology Division (CTD), Structures Laboratory (SL), US Army Engineer Waterways Experiment Station (WES), under contract to Sandia National Laboratories (SNL), Albuquerque, New Mexico. Mr. John Stormont of SNL was Technical Monitor of the field and laboratory studies reported herein, which occurred between May 1985 and April 1986, as specified in SNL Document 32-8741.

Members of the staff of the CTD Grouting Unit, under the technical supervision of Mr. John Boa, Jr., carried out the field activities. Mr. Donald M. Walley and Dr. Lillian D. Wakeley directed the laboratory development work and field activities for the CTD, with assistance from Messrs. Brian Green, Percy Collins, Donnie Ainsworth, Larry Crittenden, Cliff Gill, and Bill Neeley. The work was under the general supervision of Messrs. Kenneth L. Saucier, Chief, Concrete and Evaluation Group of the CTD; Richard L. Stowe, Chief, Materials and Concrete Analysis Group of the CTD; John M. Scanlon, Chief, CTD; and Bryant Mather, Chief, SL. Dr. Wakeley was Principal Investigator. She and Mr. Walley prepared this report.

COL Allen F. Grum, USA, was the previous Director of WES. COL Dwayne G. Lee, CE, is the present Commander and Director. Dr. Robert W. Whalin is Technical Director.

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CONVERSION FACTORS, NON-SI TO SI (METRIC)
UNITS OF MEASUREMENT

Non-SI units of measurements used in this report can be converted to SI (metric) units as follows:

Multiply	By	To Obtain
Fahrenheit degrees	5/9	Celsius degrees or Kelvins*
feet	0.3048	metres
inches	25.4	millimetres
pounds (force) per square inch	6.894757	kilopascals
pounds (mass) per cubic foot	16.018463	kilograms per cubic metre
pounds (mass) per gallon (US liquid)	80.51963	kilograms per cubic metre
fluid ounces	29.574	cubic centimetres
inches	2.54	centimetres
darcy	9.87×10^{-9}	centimetres squared

* To obtain Celsius (C) temperature readings from Fahrenheit (F) readings, use the following formula: $C = (5/9)(F - 32)$. To obtain Kelvin (K) readings, use: $K = (5/9)(F - 32) + 273.15$.

DEVELOPMENT AND FIELD PLACEMENT OF AN EXPANSIVE SALT-SATURATED CONCRETE (ESC) FOR THE WASTE ISOLATION PILOT PLANT (WIPP)

INTRODUCTION

Objectives

The purpose of this report is to describe development in the laboratory of the expansive salt-saturated concrete used for field tests at the WIPP in 1985 and 86, and explain the process of identifying variables in proportioning, mixing, placement, and curing that have potential significant impact on its field performance. Further, it describes this development process and some aspects of subsequent field placement activities from a materials science point of view. Properties of the fresh concrete are reported, as is expansion of specimens cast in the laboratory prior to field placement.

Background

Acceptance of bedded salt as a viable medium for a permanent repository for radioactive wastes first requires demonstration that in such a repository it is possible to isolate the wastes from the biosphere. Development of cementitious mixtures for use in the eventual sealing of man-made penetrations associated with geologic repositories for radioactive wastes has continued at the Waterways Experiment Station (WES) since 1975, in cooperation with Sandia National Laboratories (SNL). During this developmental period, the main objective has been to design seal materials which will be durable and long-lasting, and at the same time fairly easily emplaceable and inexpensive. Discussions of the justification for this work, and result of small-scale and laboratory testing of several candidate grouts and concretes, appear in many previous reports from WES (Wakeley and Burkes 1985; Buck 1985; Buck et al. 1985; Buck et al. 1981) and SNL (Gulick et al. 1980a,b, 1982), and in other publications (Roy et al. 1983, 1985; Wakeley and Roy 1983, 1985).

Ongoing research on cement-based materials for underground disposal of defense-generated radioactive wastes has sought combinations of materials to seal the man-made openings in rock up to a mile underground where wastes will be placed, and the shafts and boreholes that provide access to the repository horizon. Extensive work has focused on the special demands of the WIPP in southeastern New Mexico, operated by the DOE for research on and development of technology for isolation of defense-generated high-level and transuranic (TRU) wastes, in beds of rock salt. Although the salt will creep inward and eventually force the plug and rock together, the volume reduction of typical concrete following hydration could keep an opening at the interface for some

time. Such a mechanically closed interface is presumed to be potentially a less effective seal than an interface which was tight from the time of final set of the concrete. In addition, borehole plugs and shaft seals also will extend through beds of other rock types.

Concrete used at this site should seal man-made openings in the rock, and remain an impenetrable barrier for hundreds of years. Formulations based on expansive cements were expected to effect tight seals almost immediately for experimental purposes. Expansive cements were originally intended to be used under restraint, to minimize tensile stresses caused by thermal or drying shrinkage. At the WIPP, this concrete will be restrained by the rock of the repository walls.

Several reports summarize WIPP site geology and specific characteristics of the repository horizon, 2150 ft (655 m) underground (Powers et al. 1978; Stormont, 1984). Constraints on properties of mixtures for use at the WIPP stem partially from characteristics of the rock at the site, especially at the repository horizon. The mixtures must be salt-saturated, to be chemically compatible with halite host rock, particularly at time of placement when fresh mixing water would dissolve rock and enlarge the opening into which the concrete was placed (Wakeley and Roy 1983). Also, because of the need for remote placement, the concrete must have a long working time, and be fluid enough to drop through pipes or be pumped where needed.

Although it is a necessary condition, saturation of the concrete with sodium chloride leads to development of crystalline hydrated phases that are undesirable in usual concrete practice. The chemical behavior and long-term stability of these phases are less well known than are those of more usual cementitious mixtures. Special difficulties in physical handling and methods of characterization of salt-saturated cementitious compounds have complicated research on these materials from the outset. More recently, the high levels of expansion and extremely low permeabilities of candidate materials have further complicated laboratory testing. Much of the work has required modifications to standard laboratory practices or development of new procedures for mixtures that corrode steel and are partially water soluble.

Accomplishments

An expansive salt-saturated concrete was developed during FY 85 for experiments to develop technology for underground disposal and isolation of defense-generated radioactive wastes. This concrete performed better than

cement-based materials previously considered to be adequate. It was accepted for repeated in situ tests at the WIPP during FY 85 and FY 86, and may be used in conjunction with actual waste disposal, scheduled to begin in 1989.

The new salt-saturated concrete has a much longer working time and higher levels of expansion than any previously developed. It bonds readily to rock salt, and in comparison to more conventional concretes has much lower permeability when set. Concrete of this type, intentionally saturated with both sodium chloride, for chemical compatibility with rock salt, and calcium sulfate, for expansivity, is unknown in routine concrete practice.

Developing the ESC Mixture

FY 85 changes in mixture specifications

Plans for field testing the performance of vertical seals late in FY 85 and horizontal seals in FY 86 called for a salt-saturated concrete mixture with previously unachieved levels of both workability and expansion. Specifications for a concrete developed for the Office of Nuclear Waste Isolation (ONWI) in 1983 had required only a slump of four to six inches, two hours after mixing (Buck 1985). Previous field experience strongly indicated that a longer working time would be required to transport the fresh concrete from the surface to the test horizon, place it in the instrumented and non-instrumented test holes, and cast a large number of specimens (142) in various molds to be transported to WES for testing. The working time estimated for this job was over three hours, with workability indicated by a concrete slump (measured by ASTM C 143*) of greater than seven inches.

The need to increase expansive potential grew from an emphasis on early-age testing of permeabilities of seals and interfaces between concrete and host rock in SNL plans for SSSPT (Stormont, 1985). An expansive mixture giving a volume never less than the original and maintained for at least several months appeared to be the best way to assure adequate early sealing for the field experiment scheduled for July 1985. This level of sustained expansion had not been attained with previous candidate concrete mixtures (Buck 1985),

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* Annual Book of ASTM Standards, Philadelphia, PA.

nor is it usually attained with commercial shrinkage-compensating cements (Hoff and Mather 1980).

Formulation of expansive salt-saturated concrete (ESC)

ESC is an entirely new mixture, development of which was completed in June 1985, after several years of work on expansive formulations intended to be compatible with halite rock. Compatible means that it will bond to the rock, rather than dissolving it; and it will form a seal that will exclude fluid flow for some, as yet unspecified, time. Further, it will maintain a positive outward pressure on the enclosing rock at least until the rock deforms plastically to close in around it. Although it was proportioned with components intended to impart long-term durability in this special environment, assessment of its probable durability, or definition of its design service life, is outside the scope of this report.

Components of the concrete

Class H cement, similar to an ASTM C 150 Type V sulfate-resistant cement, and ASTM C 618 Class C fly ash, of high calcium oxide content, contributed to previous, moderately successful grouts and concretes saturated with sodium chloride for halite compatibility (Wakeley et al. 1985; Buck 1985; Gulick et al. 1980a,b, 1982). These materials are 15 percent of the mass of the solids in ESC (see Table 1). Additional solid components lend expansivity to this mixture: a commercial expansive admixture (essentially plaster, marketed by Halliburton as Cal-Seal®); and an expansive material marketed by Master Builders as ChemComp III (CCIII). Appendix A lists the components and reasons for including them in this mixture. Appendix B provides data from chemical and petrographic analyses of various samples of CCIII, including the one used for ESC.

Laboratory Development of ESC

Reducing water content and porosity

The need for a pumpable fresh concrete, a long working time, and yet a hardened product that was dense and impermeable necessitated delaying hydration of the cement, thus delaying the set, and using a water-reducing admixture to keep the ratio of water to cementitious solids low. A very low

air content also was expected to contribute to low permeability and high strength of the set concrete.

The need for maximum expansion also called for minimal entrained air, to keep total porosity low. Mixtures formulated to be expansive have shown expansion to decrease with increased porosity (Wakeley and Buck 1986; Odler et al. 1972): with less pore volume in which to grow, expansive phases build up more internal pressure, and enlarge the specimen. Further discussion of this subject is in the second report in this series.

Mixtures developed at WES in previous years of research for the WIPP included either melamine or naphthalene formaldehyde condensates as high-range water-reducing admixtures, meeting ASTM C 494. But in salt-saturated grouts tested during this study, use of these water-reducing and retarding admixtures had caused air contents of greater than 15 percent by volume when measured by ASTM C 231. In preparation of trial batches at WES, some of these fluid grouts had become so frothy during mixing that they exceeded the capacity of the mixer in use.

Grutzeck (1984) described similar observations from experiments with slurries of water-reducing admixtures and defoaming agents, mixed with either freshwater or NaCl brine. His work showed far less foaming with melamine formaldehyde condensates than with naphthalene. However, research at WES showed that in more chemically complex grouts and concretes, defoaming agents did not control the foaming problem, even at several times the recommended dosage and regardless of which formaldehyde condensate was used.

High air content is not a usual characteristic of concrete with a water-cement ratio as low as 0.3. From work on trying to obtain the level of air content needed for frost resistance, Whiting (1985) reported that low water-cement ratio concretes can require up to 10 times as much air-entraining admixture just to achieve air contents as high those of higher water-cement ratio mixtures. The concrete studied by Whiting (1985) had slumps in the range of 1/2 to 1 in., w/c from 0.31 to 0.36, and air contents desired to be 6.5±1.0%. The high dosages of air-entraining admixture found needed were believed due to the low slump of the concrete.

Apparently, the presence of so much sodium chloride in this mixture led to properties that were unlike those reported for more standard low-water content concretes in the unhardened state.

Increasing working
time (retarding the set)

Additional problems were encountered in retarding the set enough to give working time of three to four hours. Common commercial retarders are formulated to delay hydration of the calcium silicates and aluminates of ordinary portland cement, and thus lengthen time of setting (Young 1972). In this mixture, cement hydration was delayed whether or not organic retarders were added, by the presence of 2.5 percent (of total mass) NaCl. Although the mechanism by which salt retards the set of portland cement is not clearly understood, the fact of this effect is well documented at WES and by many others (Moore et al. 1980; Slagle et al. 1963; Hansen 1952). Use of set retarders intended to delay hydration of the calcium silicates of ordinary portland cement thus did not add to the working time of this mixture.

Prior to formulation of the expansive concrete used in the field (Table 1), trial batches involved four different chemical admixtures as virtually the only change in formulation. Appendix C gives more information about these unsuccessful attempts at formulation of an expansive salt-saturated concrete with the desired properties.

Given that the cement already was retarded by NaCl, we were unable to lengthen the working time or improve the workability of this mixture through use of any of the additives listed in Appendix C, except in cases where the resulting air content was unacceptably high. It became evident that hydration of the calcium sulfate phases, in Cal-Seal and CCIII, was preventing attainment of longer working time. In fact commercial specifications for Cal-Seal describe it as a "controlled setting gypsum cement," which sets within 20 minutes when blended with portland cement. The sodium chloride permitted a working time of more than this advertised 20 minutes, but still far less than needed.

After many experiments with various organic chemicals intended to retard the portland cement, and laboratory simulations of field conditions using these inadequate mixtures, we proportioned a concrete with the set retarder used most commonly for gypsum cements: sodium citrate (hereafter called "citrate"). As will be discussed in the two succeeding reports, the use of citrate proved to be the key to attaining the workability, low porosity, and expansive properties needed for the WIPP field demonstrations.

Optimizing Citrate Content

The ASTM standard for physical testing of gypsum concrete (C 472) requires citrate as the retarder for standard tests, at 0.2 percent by mass of sample. In concrete practice, use of citrate as a retarder for sulfate phases (hemihydrate or gypsum) is common at 0.3 to 0.5 percent by mass of cementitious solids (Sligh, 1985). With these guidelines, we prepared batches of concrete with 0.25, 0.35, and 0.5 percent citrate (by mass of cementitious solids), with no other water-reducing or set-retarding admixtures, to determine the effects of the citrate on workability and air content. The amount of defoaming agent was kept constant in these trials. We also tried using citrate without a defoaming agent, but this caused the air content to rise above 7 percent (by volume), so use of the defoamer was maintained in all other formulations. Data from these trial batches appear in Table 2.

In laboratory tests, the salt-saturated concrete with 0.5 percent citrate maintained a slump of 10 inches for nearly three hours of continuous mixing, and the air content remained less than 2.0 percent. It is this mixture to which we limit use of the term "ESC", to keep from confusing this concrete -- used in the field at the WIPP -- with all of the variations tried in the laboratory (Table 2 and Appendix C), and used for various simulations of expected field conditions (Wakeley and Poole, 1986; Pace, in prep.).

Other Properties Explored During ESC Development

Rheology

As observed during the series of slump tests on ESC, the concrete remained free flowing, with no segregation of aggregate from paste toward the periphery of its flow. This characteristic was critical, considering that the proposed emplacement method for Series A borehole seals involved dumping the concrete in several stages, dropping it through various pipes, and eventual free-fall for distances of up to 12 feet into boreholes as small as six inches in diameter (Stormont 1986b). Another property that implied the likelihood of successful placement of this concrete was adhesion of the paste to surfaces, such as the inside of the concrete mixer and of the slump cone. This behavior was judged essential to establishing an unbroken seal along the plug-rock

interfaces, and was proven in a field simulation test in January 1986, discussed later in this report.

A more exacting indication of workability can be derived from tests using a two-point workability, or Tattersall, apparatus. This test is based on the theory that fresh concrete behaves as a Bingham fluid, following:

$$\tau = \tau_o + \mu \gamma$$

where τ is the shear stress, τ_o is the yield value (minimum stress below which no flow occurs), μ is the plastic viscosity, and γ is the rate of shear. Through laboratory experiments using a Hobart mixer, Tattersall discovered that plotting the value of torque required to run the mixer vs. speed of the mixer gave a straight line, similar to that of a Bingham fluid. From this, he reasoned that the torque value (T) of a rotating impeller, in arbitrary units, represented shear stress; the speed of rotation (N) in revolutions per second represented shear rate; the intercept of the X-axis (g) represented the yield value, and the reciprocal of the slope of the line (h) gave the plastic viscosity of the concrete. The yield value (g) may indicate cohesion of the concrete, and plastic viscosity (h) is interpreted as a function of workability (Tattersall and Banfill 1983), giving:

$$T = g + hN$$

Figure 1 shows results of Tattersall tests in the laboratory on ESC. Lines intersecting the X axis on Figure 1 are best-fit curves, each defined by seven data points representing different speed settings for the impeller during a short test interval. Each curve can be thought of as representing a fixed time after mixing. Time of test (following mixing at 09:50), correlation coefficients, and slope and intercept values are listed on the figure for each curve.

The first test, represented by the left-most curve on Figure 1, was conducted one hour after mixing. Runs were repeated every 15 minutes for the second hour, and every 30 minutes thereafter. The first four curves show an essentially constant slope and intercept, indicating constant workability and cohesion. Curves representing later tests have intercepts further to the right on the X axis, indicating gradually increasing cohesiveness, although

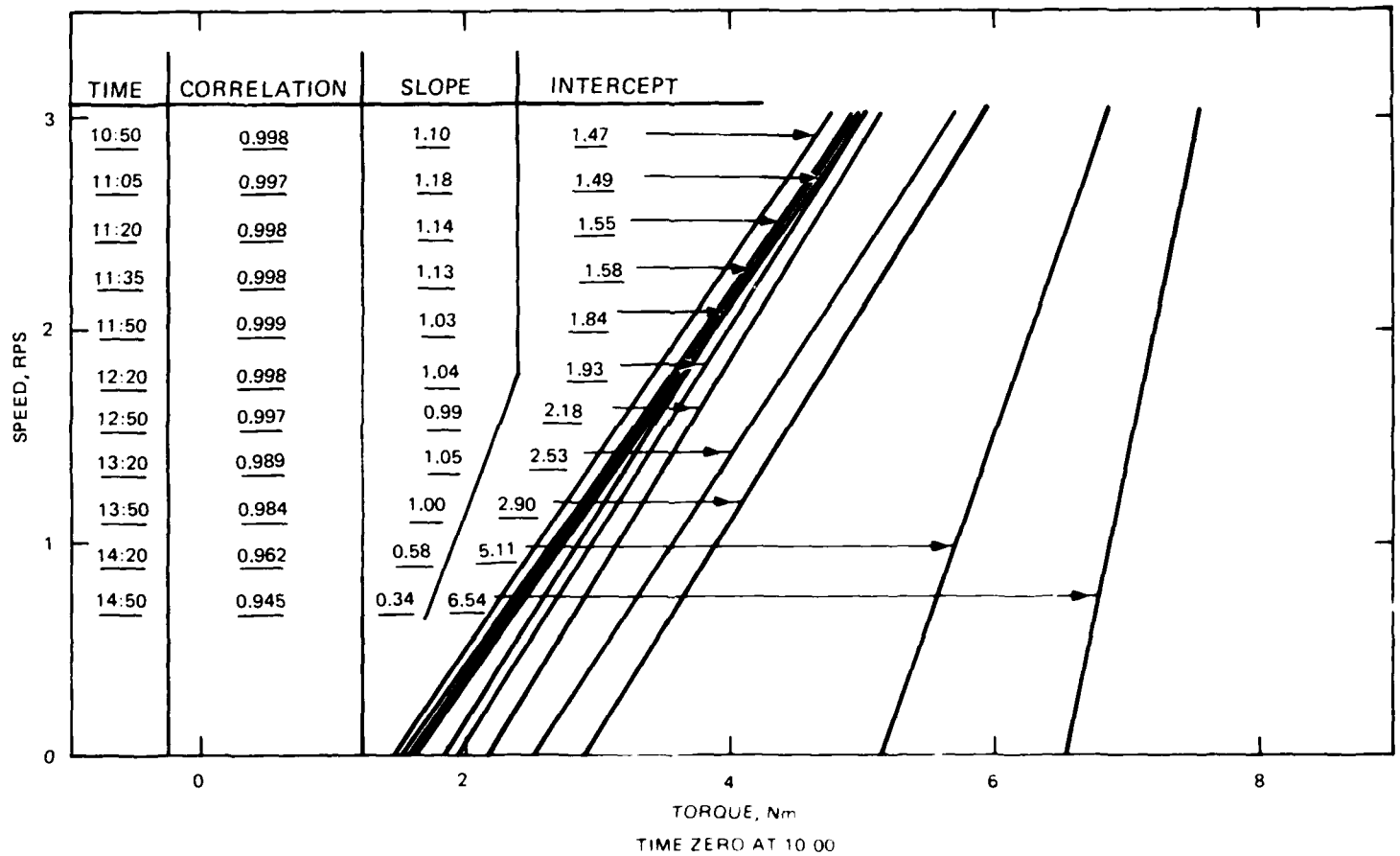

Figure 1. Two-point workability of expansive salt-saturated concrete.

the workability (1/slope) diminishes very little over the first three hours. This corresponds well to the previously described slight decrease in slump, while maintaining self-levelling flow.

The last two curves (on the right, Figure 2) represent tests at 4.5 and 5 hours after mixing, when the concrete has stiffened considerably (as shown by the location of the X intercepts). The very steep slopes of these lines probably indicate that the impeller was rotating without moving the mass of the concrete (like digging a post hole). Again, this agrees well with observed field behavior, timing of slump loss, and pumpability, and is an unusually long period of workability.

Heat evolution

Data from continuous recording of temperature in the laboratory during tests of various trial concretes consistently showed a major exotherm at 8 to 12 hours of hydration, and a second, broader peak sometimes as late as the third day after mixing (Wakeley and Poole, 1986; Pace, in prep.). Both peaks were about 88 F (31.1 C). Although this heat profile was not documented for ESC before Series A field placement, data from instrumented plugs in the field also showed a similar pattern of heat evolution, with the second maximum within 72 hours from placement.

In subsequent laboratory experiments with ESC, however, this pattern was not always observed. In at least one such test, a maximum temperature of just over 90 F occurred between 13 and 18 hours, after which the temperature dropped by 5 F during the next 50 hours, and subsequently rose to near 90 F again at four days from mixing. Temperature changes in some cases were so small and gradual that maxima hardly were recognizable as "peaks".

This characteristic pattern of heat evolution for cement hydration was recognized by Lerch in 1938, and reported in numerous publications since that time (e.g. Huang Cheng-yi and Feldman, 1985; Gotsis and Roy 1984; Lea 1971; Steinour 1952). (Actually, there is another characteristic exotherm of much greater magnitude within the first few minutes after mixing, not monitored in these experiments). The first of the near-equal peaks noted in this work is attributable to normal formation of calcium silicate hydrates in a retarded state. For most concretes, the second is attributed to delayed rapid hydration of calcium aluminate in portland cement, when the usually small amount of gypsum is depleted.

Previous studies have reported that the magnitude of this second peak diminishes, and it eventually disappears, with increased gypsum content in the mixture (Steinour 1952.) The fact of the double exotherm for ESC, then, is notable in that the second peak persists, even though diminished, in a system with this much calcium sulfate present. The peaks represent two different events of hydration, but they may be attributable to two episodes of delayed hydration, corresponding to separate retardation mechanisms for the portland cement phases and calcium sulfates, rather than to gypsum depletion. The rate and magnitude of heat generated may be diminished because the two separate systems also are reacting with each other to produce ettringite, rather than being totally independent.

In this study, the magnitude and rate of heat evolution respond not only to such factors as specimen size and rate of heat transfer away from the hydrating cement (Gotsis and Roy 1984), but also to much less obvious variables. Fairly small changes in temperature of the fresh concrete, type and amount of retarder used, and other variables of mixing and placing have a pronounced effect on thermal characteristics. The conflicting influences of these variables cause the contrasts observed in different temperature profiles of essentially the same mixture, when mixed, handled, placed, or cured in slightly different ways. The impacts of retardation, mixing, and placing variables on properties of the hardened concrete are explored more fully in the second and third reports in this series.

The decision to use iced mixing water when preparing ESC for field placement at the WIPP grew from the desire to reduce initial thermal expansion. This served several purposes. It reduced initial, thermally caused compressive stresses at the interface, which in turn diminished the resultant tensile stresses from cooling (it did not need to cool as much if it never got very warm). This probably provided a more accurate diagnosis of chemically caused expansion and resultant stresses at the interface. Also, effective working time of the concrete increased, because hydration reactions occurred more slowly at lower temperature.

Expansion

One of the properties essential to intended field performance of the ESC is a high level of chemically based (as opposed to thermal) expansion, beginning as soon as the mixture hardens and continuing at least until plastic deformation of the host rock effects a sealed interface. Previously tested

salt-saturated concretes achieved only low levels of expansion, if any (0.016% at 7 days, by ASTM C 878, and then only if specimens were cured in a fog room (at >95% R.H. and 73 F). Although this curing followed a standard method, it placed the concrete in an environment that was grossly different than known ambient conditions underground at the WIPP, where the humidity is usually between 40 and 50 percent, and temperature remains a constant 81 F, and there is no external source of water.

We decided in June 1985 to abandon standard fog-room curing in favor of an environment that more closely simulated field conditions. On June 26, about a month before Series A field placement, we cast specimens of ESC in the laboratory for measurements of restrained expansion. Specimens were as described in ASTM C 878, except that they were coated with a liquid membrane-forming curing compound on the exposed face, and kept in air at 78 F for the first 24 hours. Following this, they were demolded and coated in wax twice, with foil between, to prevent gain or loss of moisture.

Even without the standard high humidity used previously for curing, specimens of ESC reached 0.08% expansion by 7 days, 0.11% by 28 days, and 0.12% at 90 days age. Expansion data from these original laboratory-cast specimens of ESC are summarized in Table 3. Values are about twice those measured for previously reported salt concrete (Buck 1985).

Taking ESC out of the Laboratory

The decision to use ESC at the WIPP for SSSPT Series A was based on the extraordinary properties of the fresh concrete, especially its long working time, and its impressive expansive properties. Because of the change to citrate, ESC had less than 1.5% air content, indicative of anticipated low porosity and high strength. It maintained a ten-inch slump for over three hours, with a ratio of water to cementitious solids of only 0.3. It achieved the best-suited blend of materials properties yet achieved to meet the demands placed on concrete for the WIPP.

ESC PROPERTIES FROM A MATERIALS SCIENCE VIEWPOINT

Field Placement: Test Series A

When this report was completed, ESC had been prepared in Carlsbad,

New Mexico, by a commercial ready-mix company under WES supervision, on three occasions. The first of these was field placement of the concrete underground at the WIPP for Series A of the SNL SSSPT, in July 1985. Appendix D describes some of the field preparation activities at WES shortly before this event.

Stormont (1986a,b) described Test Series A in detail. In summary, ESC was placed in six vertical (down from floor level) boreholes, two each of three sizes: 6-in diameter, 1-ft length; 16-in. diameter, 2-ft length; and 3 feet, diameter and length. One of each size contained structural instrumentation in the concrete and rock, the other did not. Concomitant with field placement, 102 specimens of various configurations were cast underground at the WIPP, and returned to WES for testing of such properties as expansion, compressive strength, static and dynamic modulus, and creep. These data are reported elsewhere (Comes et al., in prep.).

The concrete was mixed at the surface, and transported to the boreholes underground through a series of buckets, pipes, hoppers, and scoops. A tremie pipe, with the lower end submerged in the freshly placed concrete, was used for filling the 3-ft holes, to minimize the potential for damaging the instruments. Finally, the concrete was consolidated in place, with internal vibrators.

This complex placement method required that the concrete be repeatedly disturbed during the approximately 30 minutes from between the time it left the mixer and when it was emplaced. The time interval was longer and the number of disturbances greater for the specimens to be transported to WES, which were prepared last. The elapsed time between introduction of water to the dry-blended ingredients -- starting hydration reactions -- and preparation of the last specimen was 3 hours and 51 minutes. Properties of the fresh concrete, and a summary of the timing of some of the major steps in placement procedures, are given in Table 4.

Data from the first 28 days of monitoring expansion of specimens cast with Series A concrete showed reduced restrained expansion relative to their laboratory-cast counterparts. This led us to hypothesize that the time required and techniques involved in placement had some effect on the expansive potential of the set concrete. A systematic testing of this hypotheses is the main subject of Report 2 in this series.

Simulation of Horizontal Placement

Plans for Series B of the SSSPT required that the concrete be placed in horizontal boreholes. In January 1986, the second commercial mixing of ESC took place, when WES staff members conducted a test of horizontal placement using a clear plastic pipe 3 feet in diameter (Figure 2). For this test, ESC was placed with a concrete pump, proving that pumping was a viable technique for sealing horizontal openings.

This simulation also demonstrated several other beneficial placement properties of ESC, which had been suggested in laboratory operations but not directly observed. The clear plastic pipe permitted observation of the flow behavior of the fresh concrete, which was seen to flow easily around the instrumentation support structure (a mock-up with no gages). The mixture was fluid enough to be self levelling; and the aggregate was seen to remain evenly distributed, without segregation from the paste, which adhered readily to the pipe wall. Also, the concrete did not penetrate very far into a bed of crushed salt, held in place by screening at one end of the pipe and necessary for permeability tests (Wakeley and Poole 1986; and Stormont 1986a) and to preclude end effects (Stormont 1986a).

The freshly placed concrete was pressurized by small increments, so that its behavior under pressure could be observed. At 50 psi, however, the plastic pipe failed at its bulkhead seal and the concrete flowed out. This was unfortunate, because it precluded direct observation of bond between pipe and concrete, and of expansion after set.

Series B Field Placement

The second major field event of the SSSPT occurred on February 28, 1986, at the WIPP. The three boreholes plugged during Series B all had 36-inch diameters. Two were instrumented similarly to those in Series A (Stormont and Howard, 1986), and all were scheduled for test of in situ permeability. The difference from Series A was that these holes were drilled horizontally into a thick bed of rock salt at the test horizon, and fronted with bulkheads.

The change from vertical to horizontal configuration required changing the method of concrete placement from gravity-aided freefall to pumping. The successful placement of Series B borehole plugs confirmed the feasibility of

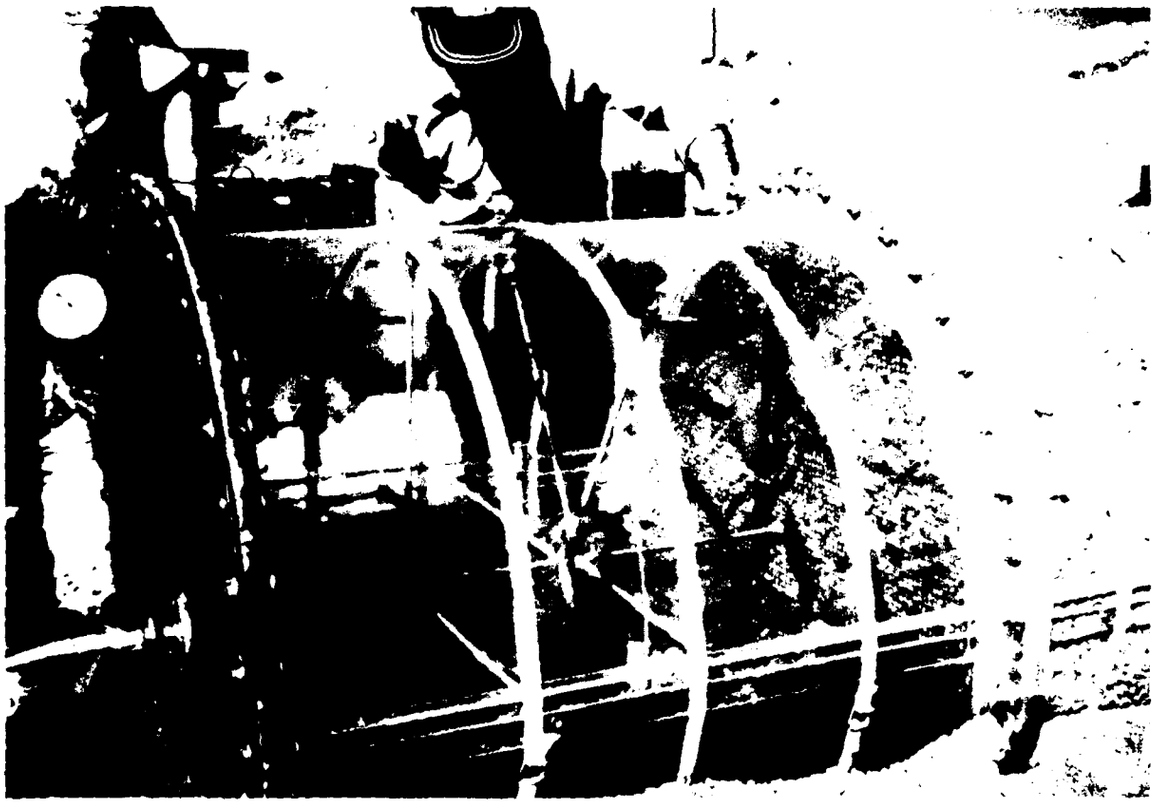

Figure 2. Test of pumping ESC into horizontal borehole, simulated by plastic pipe, January 1986.

reproducible batching of this complex concrete mixture with commercially available facilities. Properties of fresh ESC as measured in the field for this event appear in Table 5.

As part of Series B field activities, 141 specimens were cast underground at the WIPP and either trucked directly or air-shipped to WES, for testing beginning at three days age. All of these specimens were cast from concrete that had been through the pump. Pumping had no apparent deleterious affect on properties of this concrete as indicated by laboratory tests, although expansion of the field-cast specimens was not as great as that of the original laboratory-cast ESC. Selected measurements of physical properties from field-cast specimens tested at early ages appear in Table 6. The bulk of these data from early-age testing appear in another report (Comes and others, in prep.).

SUMMARY AND RECOMMENDATIONS

An expansive salt-saturated concrete formulated at WES meets or exceeds the goals of workability, low air content, and high early and sustained expansion for use underground at the WIPP. Hydration of the portland cement phases of this concrete is retarded by the sodium chloride necessary for chemical compatibility with the host rock. Hydration of sulfate phases, especially the plaster component, is retarded by sodium citrate. Even without additional organic chemical water reducers or set retarders, the mixture has a low water content and working time of up to four hours. Despite its complexity, this concrete can be reproducibly batched with commercially available facilities, and can be placed by either gravity placement or pumping.

A comparison of expansive properties of laboratory-cast vs. field-cast specimens suggested that there are several factors of mixing and placing that affect the level of expansion, and thus have an impact on field use of this mixture. Those that we identified during mixture development and characterization include:

(a) temperature of the mixing water, and of the mixture before hardening;

(b) curing temperature;

(c) amount of citrate used;

(d) time delay between mixing and casting; and

(e) disturbance or quiescence during this time delay.

We have explored these factors in laboratory experiments, and report them in the second Technical Report in this series (Wakeley 1986).

In a significant departure from routine practice, we adopted curing conditions for specimens from both laboratory and field casting that closely simulate conditions of the WIPP repository horizon. Specimens are sealed to prevent moisture gain or loss, which we judged to be similar to their being encased in rock salt. They are then maintained in air at 81 F, instead of in conditions specified in ASTM standards (fog room at 73 F).

The behavior of this expansive mixture is grossly different in these two sets of conditions, with the standard methods unrelated to real demands on concrete performance. We recommend that field conditions be simulated for curing any such experimental and special-purpose material, especially for any salt-saturated and highly expansive concretes, which are quickly degraded at high humidity.

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Table 1

Components and Proportions of Expansive Salt-Saturated Concrete (ESC)

Component[a]	% of total by mass	% of total solids, by mass	Actual batch weight, lbs[c]
Class H cement	9.03	9.66	1822.5
Chem CompIII	6.02	6.45	1215.0
Cal-Seal	1.80	1.94	364.5
Class C fly ash	5.10	5.44	1026.0
Fine aggregate	34.11	36.50	6823.6
Coarse aggregate	34.58	37.00	6903.6
NaCl	2.50	2.65	499.5
Defoaming agent	0.21	0.24	42.9
Na citrate	0.11	0.12	0.164
Water (iced)	6.60	--	1461.7
Total	100.06	100.00	

a) Sources of components and additional information on selection and use are in Appendix A.

b) Aggregate total is 61.1% by volume of fresh concrete.

c) Batch prepared for Test Series A of the SSSPT.

Table 2

Set-Retarding Admixtures Tested as a Component of Salt-Saturated Concrete[a]

Admixtures[b]	Problems Encountered in Use[c]
1. Melgran "0"	rapid increase in slump over 2 hours
2. HR-12	ineffective as set retarder
3. CFR-1	extreme set delay (days to weeks in low doses)
4. Daracem 100	high air content; ineffective as retarder

[a] Other components of the concrete remained constant, and are as listed in Table 1. These admixtures were used instead of sodium citrate during development of ESC. There were slight adjustments in water content and amount of retarder used.

[b] Additional information about concrete formulations with these admixtures is provided in Appendix C.

[c] This is not a judgment of the effectiveness of these products in conventional concrete practice. The problems listed here are a result of this mixture containing large amounts of both sodium chloride and calcium sulfate.

Table 3

Restrained Linear Expansion of Original Laboratory-Cast Prisms of ESC (06-26-85)

Age, days	% linear expansion[a]
2	0.029
5	0.044
7	0.084
14	0.113
21	0.110
28	0.111
56	0.127
90	0.124
180	0.118
270	0.120

[a] Average of two specimens

Table 4

Timing of Field Activites and Properties of Fresh ESC,

SSSPT Test Series A, 30 July 1985

Time, AM	Time Lapsed h:min	Event	Mixture Properties
0452	0:00	Water (@ 34°F) introduced to dry-batched solids.	
0528	:36	First bucket of ESC lowered down shaft toward repository horizon.	Temp: 81°F Slump: 10-1/4 in. Air content: 1-1/2%
0709	2:17	Fifth (last) bucket of ESC down shaft	Temp: 76°F Slump: 9-1/4 in.
0758	3:06	Filled boreholes in L-1 alcove	Temp: 80°F Slump: 8-3/4 in.
0843	3:51	Finished casting test specimens in L-2 alcove	Slump: 3-3/4 in. Air content: 2.0%

Table 5

Timing of Field Activites and Properties of Fresh ESC,

SSSPT Test Series B, 28 February 1986

Time	Time Lapsed hr:min	Event	Mixture Properties
0910	0:00	Iced mixing water introduced to dry-batched solids.	
0933	:23	Concrete tested before first bucket sent downhole.	Temp: 56°F Slump: 10-1/2 in.
1023	1:13	First batch of concrerte through pump at test horizon.	Temp: 60°F Slump: 9-1/2 in. Air Content: 1.8%
1052	1:42	Concrete tested again before last bucket sent downhole.	Slump: 9-3/4 in.
1220	3:10	Began making specimens for laboratory tests.	
1246	3:36	Tested concrete from last bucket through pump.	Temp: 64°F Slump: 8-1/2 in.
1333	4:23	Finished casting test specimens.	

Table 6

Restrained Linear Expansion of Standard (ASTM C 878) Prisms of ESC from Series A and B Field Events

	% Linear Expansion	
Age, days	Series A[a]	Series B[b]
2	0.010	0.026
3	0.030	--
4	--	0.059
6	0.042	0.084
7	0.029[c]	0.086
10	0.032	0.089
14	0.033	0.089
21	0.036	0.092
24	0.036	0.093

[a] Cured @ >80°F in the field for five days, then @ ~74°F in the laboratory; sealing from moisture loss was inadequate.

[b] Cured continuously @ 81 to 85°F, sealed to prevent moisture loss.

[c] Moved to cooler curing conditions; apparent shrinkage attributed to thermal rather than chemical change.

APPENDIX A

COMPONENTS OF ESC AND REASONS FOR THEIR USE

APPENDIX A: COMPONENTS OF ESC AND REASONS FOR THEIR USE

A. Most components of ESC have been identified, through several years of development of grout and concrete for the WIPP site, as having characteristics considered beneficial to cement-based materials for use in the Plugging and Sealing Program. Use of sodium citrate in these materials has the shortest history, beginning with ESC in June 1985. ESC components are:

1. Class H oil-well cement, chosen for coarse grind, low water demand, sulfate resistance, availability, and long history of successful commercial use.

2. Class C fly ash (ASTM C 618) with high calcium oxide content, supplied by Southwest Public Service. It was chosen for its anticipated contribution to chemical expansion, availability at a reasonable distance from the WIPP site, and uniform composition over time.

3. Cal Seal (a trade name for calcium sulfate hemihydrate) and ChemComp III both were intended to contribute to the expansivity of the concrete, and give favorable results when used with Class H cement and high-lime fly ash.

4. Granular sodium chloride is added so that the mixture is saturated with this salt, and can be placed in contact with halite rock without dissolving the rock at the interface. Secondarily, it contributes to the workability and expansiveness of the mixture.

5. De-Air #1 is a proprietary air-detraining agent of Halliburton Services, Inc. In conjunction with other admixtures used in ESC, this component allows mixing for up to three hours while keeping air content below 3 percent.

6. The sand and gravel used as aggregates in ESC are commercially available near the WIPP site, and had no noticeable deleterious effects on the concrete.

7. Sodium citrate acts as both a set retarder and a water reducer in this concrete, and is used in lieu of other, more recently developed and marketed organic chemical admixtures for these purposes.

8. Proportions of these components used in ESC are given in Table 1 of this report. Additional information about admixtures appear in Appendix B.

APPENDIX B

PETROGRAPHIC EXAMINATIONS OF EXPANSIVE ADDITIVE RC-925 (CHEMCOMP III)

Memorandum for L. D. Wakeley, Materials and Concrete Analysis Group,
Concrete Technology Division, Structures Laboratory.
Prepared by J. P. Burkes dated 3 Sep 85.

Background

1. ChemComp III is an expansive additive currently being used in several programs in which large amounts of expansion are required. Variable results from different shipments of this additive prompted investigation to determine if compositional variations were causing differences in the expansion developed in various concrete mixtures. The latest shipment of ChemComp III was received in March 1985 (RC-925). The phase composition of this sample was compared to two earlier shipments of ChemComp III designated RC-898(2) and RC-900.

2. X-ray diffraction (XRD) patterns of all three additives suggested that the composition of the latest shipment of ChemComp III differed from that of the two earlier shipments. A detailed petrographic examination of the latest shipment was made to provide information concerning the following questions:

a. What are the differences in the three batches of ChemComp III?

b. What, if any, filler is present in RC-925?

c. Does RC-925 contain any portland cement?

d. What is the composition of the additive, especially particles with specific morphologies?

Sample

3. Approximately 700 g of RC-925 were received in May 1985 from L. Wakeley. The sample was sealed in a plastic bag.

Test procedure

4. The as-received sample was examined by XRD, scanning electron microscopy (SEM), energy dispersive X-ray (EDX) analysis, and with a polarizing microscope. The sample examined by XRD was a tightly packed powder. No additional grinding was done. It was X-rayed in a static nitrogen atmosphere to minimize hydration during the XRD scan.

5. Examinations of the material by SEM and EDX were made on specimens prepared by two different methods. One method consisted of fabricating a pressed pellet of the material forming a plane surface. This method provided the best input data for elemental analysis using EDX.

6. To provide better particle discrimination by morphology, a specimen was prepared for SEM examination by using an ultrasonic cleaner to disperse a small portion of the sample in ethyl alcohol. A drop of the dispersed

material was placed on a carbon mount. When dry, the sample was coated with a layer of carbon (approximately 50 Å thick) and then a thicker layer of gold (approximately 100 Å).

7. A grain immersion mount was prepared using immersion oil with a refractive index of 1.544 for examination using a polarizing microscope.

8. Selective dissolution. Five grams of as-received RC-925 were treated for 30 minutes with a 20 percent solution of maleic acid and methanol. This procedure dissolves calcium silicate phases such as alite and belite. The weighed residue was examined as follows:

a. A tightly packed sample was examined by XRD.

b. A small portion of the residue was dispersed in ethyl alcohol and examined by SEM and EDX. The specimen preparation was similar to that described earlier.

c. A grain immersion mount was prepared as before and examined with a polarizing microscope.

9. Two grams of as-received sample were treated for 2 hours in a 10 percent solution of ammonium chloride (NH_4Cl). This treatment dissolves sulfate phases. It removed gypsum and calcium sulfate hemihydrate from the sample. The NH_4Cl treatment also removes alite and belite but does not affect the tetracalcium trialuminate sulfate ($C_4A_3\overline{S}$) phase. The residue from this treatment was examined as described earlier for the maleic acid residue.

10. A laboratory prepared sample of $C_4A_3\overline{S}$ was examined by XRD. This XRD pattern was compared with selected peaks in the XRD patterns of RC-925.

11. One of the elements found in the residue after NH_4Cl treatment was verified by analysis using an inductive coupled plasma spectrometer (ICP). Analyses were made on an as-received sample of RC-925, the NH_4Cl used in the dissolution treatment, and the RC-925 residue following NH_4Cl treatment.

12. All XRD examinations were made with an X-ray diffractometer using nickel-filtered copper radiation.

Results

13. Eight or nine crystalline compounds were identified by XRD. The calcium silicates alite and possibly belite were present. These compounds were identified by the presence of weak 6.1-, 4.7-, 2.74-, and 2.197-Å peaks and stronger peaks at 2.8 and 2.76 Å. The other compounds found were tricalcium aluminate (C_3A), an aluminoferrite solid solution phase, magnesium oxide (MgO), gypsum ($CaSO_4\ 2H_2O$), calcium sulfate hemihydrate ($CaSO_4 \cdot 1/2H_2O$), C_4A_3S, and quartz. The presence of noncrystalline material was indicated by a halo on the XRD pattern made from the NH_4Cl treated sample. A 3.18-Å peak was present in the NH_4Cl treated sample; it was not identified.

14. The amounts of the calcium silicates and calcium sulfates present were determined by the selective dissolution techniques that were used. Calcium silicates were calculated to be 33.5 percent of the sample while gypsum and hemihydrate were calculated to be 29.2 percent.

15. Fifty-one EDX spectra and 12 SEM micrographs were made during analysis and examination of the untreated sample and the two insoluble residues. Ten EDX spectra are included as Figures 1 through 10. Five of the twelve SEM micrographs are included as Micrographs 1 through 5. Figure 1, the EDX spectrum of a pressed pellet, and Figure 5, the EDX spectrum of gypsum, did not have corresponding micrographs.

16. In general, the results of the SEM and EDX work are as follows:

a. The elements shown by EDX analysis of a composite pressed pellet were Na, Al, Si, S, K, Ca, Ti, and Fe (Figure 1). Tin (Sn) and magnesium (Mg) were also present. However, their concentration was too low to be detected in the bulk analysis of the pressed pellet. Lead (Pb) was present in the NH_4Cl residue. Magnesium was found in some individual particles in the insoluble residues from both chemical treatments and in the calcium silicate particle from the as-received material (Figure 2). However, Sn and Pb were only found in a few of the particles from the residue after treatment with NH_4Cl. Additional analysis by ICP indicated the presence of Sn in the as-received sample. The ICP analysis is shown below in percent SnO.

Samples Tested	SnO, %
NH_4Cl reagent	0.0005
RC-925	0.005
RC-925 residue following NH_4Cl treatment	5.95

Lead was not tested for but was assumed to be coming from the NH_4Cl since the analysis provided on the container suggests "Heavy Metals (as Pb) - 2 parts per million (PPM)."

b. Most particles lacked specific morphology. Gypsum and hemihydrate were the only phases that could be identified by their morphology. The other phases were particles with rounded or irregular boundaries (Micrographs 1-4).

c. Portland cement phases were identified in the sample by XRD as well as by EDX. Micrograph 1 shows a typical calcium silicate particle found in this sample. The EDX spectrum (Figure 2) shows this grain contains K, S, Mg, and Al as impurities. Other calcium silicate particles examined also contained varying amounts of impurities. Other portland cement constituents that were tentatively identified by their EDX spectra were C_3A (Micrograph 2) and a calcium aluminoferrite solid solution phase (Micrograph 3). The spectrum (Figure 3) taken from an area of the particle in Micrograph 3 shows as major elements Fe, Ti, Al, and Ca. These major elements were the basis for tentatively identifying the particle as a calcium aluminoferrite solid solution phase. Minor amounts of S, Si, K, and Mg also were present. Tricalcium aluminate particles were identified by

chemical composition using EDX spectroscopy. The particle in the right center of Micrograph 2 was identified as C_3A. The major elements present were Ca and Al (Micrograph 2, Figure 4). The particle also contained trace amounts of Fe, Ti, S, Si, and Na.

d. Sample RC-925 contains roughly 30 percent of gypsum and hemihydrate. These phases were easily detected in the as-received and maleic acid residue samples using a polarizing microscope and SEM. Each EDX spectrum made of gypsum and hemihydrate particles showed only Ca and S since hydrogen and oxygen are not detectable. Figure 5 is a typical spectrum of one of these calcium sulfates.

e. The NH_4Cl treatment removed about two-thirds of the sample (62.7 percent). The residue contained $C_4A_3\bar{S}$ as its major phase. Tricalcium aluminate, an aluminoferrite solid solution phase, MgO, quartz, glass, and possibly calcium silicate sulfate were also identified as part of the residue. Several $C_4A_3\bar{S}$ particles were analyzed (Micrograph 4) by EDX. The elemental composition of the particles consisted of Al, Ca, and S as major constituents with smaller amounts of Fe, Si, Mg, Ti, K, and Na nearly always present (Figures 6, 7, and 8). The small particles with a crystalline appearance contained in the large particles in Micrograph 4 had more Al, Ca, and S and less of the other elements (Figure 7). The least contaminated $C_4A_3\bar{S}$ found lacked specific morphology (Micrograph 2, Figure 8).

f. A few particles found in the residue following NH_4Cl treatment had complex EDX spectra making correlation with phases identified by XRD difficult. The irregular shaped particle in the center of Micrograph 2 contains Pb and Sn. Several particles with the same morphology that showed Sn and Pb on EDX spectra were found (Figure 9). Some round particles (Micrograph 5) indicated by EDX to be totally Al (Figure 10). Particles with this morphological feature were also examined with a polarizing microscope and will be discussed later.

17. Examination with a polarizing microscope of grain mounts made from as-recieved material, residue after maleic acid treatment, and residue after NH_4Cl treatment gave results that agree with those obtained by XRD and SEM and EDX. The following are the descriptions of particles and their identifications as determined using a polarizing microscope:

a. The mount made from as-received material showed low index, weakly birefringent particles of gypsum. Much of the gypsum had characteristic rhombic forms. The hemihydrate had slightly higher index of refraction and much higher birefringence. Tricalcium aluminate was identified by its isotropic nature. Calcium silicates and the aluminoferrite solid solution phase showed their characteristically high index of refraction. The calcium silicates were transparent to translucent in plane polarized light while the aluminoferrite phase showed brownish color. The $C_4A_3\bar{S}$ is mostly isotropic. However, at about 600X magnification, birefringent areas can be seen in most particles. Some of the $C_4A_3\bar{S}$ grains appear to have glassy areas.

b. The grain mount of residue after maleic acid treatment appeared similar to the as-received sample except the calcium silicates were no longer present.

c. The examination of residue after NH_4Cl treatment showed the expected absence of calcium silicates, gypsum, and hemihydrate. Round isotropic particles with an index of refraction higher than 1.544 were found in this sample. These particles appear to be glass and show a slight greenish color. A particle similar in shape is shown in Micrograph 5.

18. The XRD data obtained from the laboratory prepared $C_4A_3\bar{S}$ agreed well with the XRD data for $C_4A_3\bar{S}$ in RC-925 (Table 1). The 13.0-A peak was common to both the RC-925 and the laboratory prepared $C_4A_3\bar{S}$ but was not shown in the JCPDS* powder diffraction card 16-335 for $C_4A_3\bar{S}$.

Discussion and conclusions

19. The petrographic examination of RC-925 identified several crystalline phases plus some glass. Portland cement minerals alite and possibly belite (calcium silicates), tricalcium aluminate, and a calcium aluminoferrite solid solution phase were present. Gypsum and $C_4A_3\bar{S}$ were the major phases identified. Quartz, MgO, and hemihydrate were minor constituents. No fillers were detected.

20. A small amount of calcium silicate sulfate may be present as indicated by the 3.18-Å peak in the NH_4Cl treated sample. This material has been found associated with Type K cement, and $C_4A_3\bar{S}$ is a major component in Type K cement.

21. The chemical elements found by EDX analysis of a composite sample were Ca, Ti, Fe, K, S, Si, Al, and Na. Calcium was the major element in the sample. EDX spectra of single particles showed that Mg, Sn, and Pb also were present in some particles. Tests using ICP for Sn in the NH_4Cl reagent, the as-received RC-925, and the RC-925 residue following NH_4Cl treatment suggests that the Sn is a component of the sample and not a contamination. The lead present was probably contamination from the NH_4Cl reagent used in the dissolutioning process.

22. Individual particles of a calcium silicate, C_3A, a calcium aluminoferrite solid solution phase, gypsum, and $C_4A_3\bar{S}$ were identified by their EDX spectra. Gypsum was the only phase that could be identified by its morphology. Identification of Na, Mg, K, Si, Fe, and Ti in spectra of some single particles that showed major elements of Ca, S, and Al suggest the $C_4A_3\bar{S}$ has a complex composition and that some substitution has occurred in the $C_4A_3\bar{S}$ lattice. Parts of these particles appear to be glass.

23. Isotropic particles that show only Al by EDX are present in the sample. These may be a glassy aluminum oxide. Only a trace amount of such particles was present.

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* JCPDS - International Centre for Diffraction Data, 1601 Park Lane, Swarthmore, Pennsylvania 19081.

24. Consideration of selective dissolution data, phase identifications, and limited chemical data indicate that the total sample is about 60 percent $C_4A_3\bar{S}$ and gypsum and 40 percent portland cement phases. It is not known if the cement is an integral component of manufacture or is an addition. Approximately 33 percent of the sample is calcium silicates.

25. A comparison of data obtained from this sample (RC-925) and RC-898(2) indicates they are very similar.* The phases present in RC-898(2) and RC-925 (portland cement phases and the sulfates and $C_4A_3\bar{S}$) are also present in RC-900, but the relative amounts of the individual constituents differed.

* Memorandum for John Boa, Concrete and Grout Group, Structures Laboratory, "X-ray Diffraction Examination of RC-898(2) and RC-900," 5 October 1983.

Table 1

A Comparison of Experimental XRD Data to JCPDS Data for $C_4A_3\overline{S}$

Laboratory Prepared $C_4A_3\overline{S}$ "d" Spacing (A)	JCPDS Card 16-335 Relative Intensity	RC-925
13.0		X
6.55	2	X
5.82		
5.43		
4.92	6	X
4.69		
4.61	<1	X
4.33	2	X
3.85		
3.76	100	X
3.61		
3.50		
3.40		
3.36		
3.25	8	X
3.16	2	X
2.99	2	X
2.91	8	X
2.84	2	X
2.65	25	X
2.60	2	
2.51	2	X
2.46	8	X
2.42	2	
2.338	<1	
2.297	4	X
2.261	2	X
2.192	2	X
2.166	20	X
2.135	4	X
2.055	<1	
2.029	2	
1.984	2	X
1.959	2	X
1.936		
1.876	2	X
1.857	2	X

APPENDIX C

PREPARING SALT-SATURATED CONCRETE WITH SET-RETARDING OR WATER-REDUCING ADMIXTURES OTHER THAN SODIUM CITRATE

APPENDIX C: PREPARING SALT-SATURATED CONCRETE WITH SET-RETARDING OR WATER-REDUCING ADMIXTURES OTHER THAN SODIUM CITRATE

A. Description of our experiences with the admixtures discussed here should not be taken as a recommendation for or against their use in normal concrete practice. The characteristics they imparted to this concrete were at least partly controlled by its high levels of both sodium chloride and calcium sulfate. Performance of admixtures in this unique concrete should not be extrapolated to other situations or mixtures.

B. The term "ESC" is used exclusively for the concrete defined in Table 1, with sodium citrate but without any of the admixtures listed here.

C. The admixtures we tried and the properties they imparted to the concrete are:

1. Melgran "O", a melamine-based proprietary high-range water reducer, produced by American Admixtures Co. Starting with 0.4 lb/cu ft Melgran an w/c = 0.3, slump decreased from 10.75 to 2.75 in. over the first 70 min after mixing. We added both Melgran and water, to 0.9 lb/cu ft and w/c of 0.41, but slump was still too low 2 hours after mixing, and the air content had increased from 1.2% to an unacceptable 1.8%.

2. Daracem-100 is a proprietary combination of high-range water reducer and retarder, produced by W. R. Grace and Co. Used in similar proportions, the resulting concrete had an initial slump of 11 in., and air content 2.6%. After a half hour rest an 5 min. of mixing, the slump was constant but the mixture had incorporated 6.9% air. An hour after initial mixing, the slump had decreased to 4.5 in. Adding 50% more Daracem pushed the slump up to 9 in., but the air content was over 6%.

3. CFR-1, from Halliburton Services, was used at 0.56% by weight of cementitious materials. This gave adequate slump (over 9.5 in.) to more than 2 h, and kept air content below 2% for that time. However, specimens did not set within a week after mixing, and those placed in a fog room essentially never set. Although it may have been possible, through further experimentation, to determine a dosage that would give adequate workability and a practical set time, it appeared that field use of this admixture would require more careful proportioning and measuring than is practical in field conditions.

4. When formulated with HR-12, another Halliburton product, the concrete exhibited rapid slump loss. Being ineffective as a set-retarder in this high-sulfate mixture, this product was not tested for air entrainment.

APPENDIX D

CTD INTERNAL MEMO SUMMARIZING PREPARATION FOR SSSPT SERIES A

Prepared by Dr. L. D. Wakeley, dated 19 July 85

Concrete Development and Field Preparation, Week of 15-19 July 85

1. An expansive salt concrete with 0.5% Na-citrate (by weight of cementitious solids) was prepared and tested further for aspects of its probable field performance. Workability was monitored extensively. We found this mixture to flow freely from an inverted slump cone, following 10 minutes of mixing and as much as 3 hours of aging without further mixing.

2. This mixture also exhibited no apparent segregation of aggregate in flow tests, an important consideration for a mixture the placement of which will require up to 12 feet of free fall.

3. Plexiglass pipe of 4-inch i.d. was used in the lab to simulate a borehole in the floor of the WIPP repository horizon, for tests of concrete placement methods. Equipment for funneling the concrete into boreholes of this relatively small diameter was modified as needed.

4. On 07-16, several attempts to brace PVC pipe (1/2" o.d.) against the inner wall of the plexiglass tube were unsuccessful. For permeability tests planned for the field later this month, we needed to maintain contact along the interface between rock and concrete. But PVC in 12-foot lengths is not straight, nor is it rigid enough to be held readily in place.

5. Of the five methods tried to hold the PVC in place, the only one that was even partially successful also caused bridging of the aggregate during concrete placement, preventing us from filling the plexiglass tube. All three lengths of PVC tended to bunch up on one side of the tube, and could not be held separately and still permit the concrete to pass freely down the tube. Also, it was obvious that turning such "floppy" pipe within the concrete would be nearly impossible, because of the loss of torque. We abandoned PVC.

5. The plexiglass tube was emptied and reused on 07-17, with the same concrete mixture but with rigid electrical-conduit type pipe instead of PVC. The conduit was held in place at the base of the plexiglass tube by a specially designed plexiglass spacer (as had been tried with PVC), and at the top with fiber-reinforced tape. The concrete-placing equipment was used successfully with this set-up, and the tube was filled up to about six feet.

6. Six hours after placement of the concrete, the sections of conduit were turned fairly readily, using a small pipe wrench. Each was pulled up a few inches from the bottom of the tube, to assure free movement. We pulled one of the three sections out of the tube, to see if 6 hours was adequate time for the concrete to gain enough rigidity to maintain the "hole" left where the conduit had been. It was not: the hole collapsed.

7. At 20 hours, the 2 remaining lengths of conduit were held too rigidly in the concrete to be either turned or pulled out. Repeated turning between 6 and 20 hours will be required in the field. It should be possible to remove the sections of conduit at 20 hours, however.

8. On 07-17-85, we partially filled two glass jars with the concrete that had been dormant for 3 hours, and maintained them at lab ambient conditions for curing. The jars were intact at 24 hours, but one had broken from concrete expansion by 40 hours. The second was still intact at 48 hours.

END

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DTIC